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<td>Ohmori, Akira; Shimomiya, Shougo; Kawakami, Fumio; Iwamoto, Nobuya</td>
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<tr>
<td><strong>Citation</strong></td>
<td>Transactions of JWRI. 19(1) P.107-P.112</td>
</tr>
<tr>
<td><strong>Issue Date</strong></td>
<td>1990-06</td>
</tr>
<tr>
<td><strong>Text Version</strong></td>
<td>publisher</td>
</tr>
<tr>
<td><strong>URL</strong></td>
<td><a href="http://hdl.handle.net/11094/5455">http://hdl.handle.net/11094/5455</a></td>
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Examination of Plasma Spray Parameters for YBa$_2$Cu$_3$O$_{7-x}$ Coatings

Akira OHMORI *, Shougo SHIMOMIYA **, Fumio KAWAKAMI ** and Nobuya IWAMOTO **

Abstract

This paper examines the influences of plasma spray parameters on property and structure of YBa$_2$Cu$_3$O$_{7-x}$ coatings produced by air plasma spraying using calcined superconducting YBa$_2$Cu$_3$O$_{7-x}$ powder and raw material spray dried powders of Y$_2$O$_3$, 2BaCO$_3$, and 3CuO.

As plasma spray parameters, plasma arc power, plasma gas flow rates, kind of gases and standoff distance were adopted. In order to obtain a superconducting coating uniformly and easily by appropriate thermal treatment, it was important to suppress the decomposition of powder through plasma jet, so that it was necessary to select plasma spray conditions of lower heat content for the superconducting powders. On the other hand, it was important to facilitate the mutual reaction among 3 kinds of oxides during plasma spraying and to increase heat content for the agglomerated powders.

KEY WORDS: (Plasma spraying) (Spray parameter) (YBa$_2$Cu$_3$O$_{7-x}$) (Coating) (Superconductivity)

1. Introduction

A number of investigations for processing and properties of superconducting coatings produced by plasma spray techniques have been reported. 1-12) Most of them described for properties of coatings thermally treated relatively at high temperature and for long time. However, there were a few that discussed the influences of plasma spray parameters on as-deposited compositions. To spread the applications of this process which has resulted in a number of advantages such as processing in atmosphere, high deposition rate, less restriction in thickness, shapes and so on, it is important to make problems clear for applying plasma spraying process to the formation of superconducting coatings.

On this process, it is recognized that the thermal effect is excessive, momentary and different particle by particle during plasma spraying, so that it is not easy to maintain characteristics without any decomposition of structure and disruption of phase throughout the course of deposition especially for superconducting powders. Then, a post thermal treatment has been applied to provide superconductivity. As a superconductor, generally, the coating requires high density and uniformity for good electric and magnetic properties, however, a dense coating usually requires high power operations and post thermal treatment after spraying. Therefore, considering plasma spray process as a pre-forming step, except the matter of density, and to abbreviate thermal treatment, the influences of spray parameters on transformation of compositions were investigated using superconducting powders and non-reacted agglomerated powders.

2. Materials and Experimental procedure

YBa$_2$Cu$_3$O$_{7-x}$ superconducting powders were prepared by the solid state reaction method. Agglomerated powders were prepared by conventional spray-dried method with Y$_2$O$_3$, BaCO$_3$, and CuO reagents (Y : Ba : Cu = 1 : 2 : 3). SEM photographs and XRD results of each powder are shown in Fig. 1. The deposits were produced with Metco 9MB and 3APG plasma spray torches, and formed over 0.3mm thickness on a thin blasted stainless sheet. Then it was carefully twisted for deposits to be removed from substrate and they were thermally treated at 1223K for 0min, 2hrs, 6hrs in air and cooled in the furnace. At first, the influence of plasma power, arc current and voltage respectively, were examined by using Ar mono gas as plasma and powder carrier gas. Then a kind of gas, a volume of plasma gas and standoff distance were examined.

Plasma spray conditions used in this experiment are shown in Table 1.

All deposits, sprayed and thermally treated, were

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Transactions of JWRI is published by Welding Research Institute of Osaka University, Ibaraki, Osaka 567, Japan
exmained by SEM, EDX and X-ray diffraction. And superconducting properties of thermally treated deposits were examined by resistivity measurement (Tc) carried out by a four-probe method.

3. Results and Discussion

3.1 Effect of plasma power on Tc of deposits

Figure 2 shows the effect of plasma power and thermal treatment on the XRD patterns and Tc of plasma sprayed deposits produced under plasma spray conditions in Table 1 with superconducting powders.

From X-ray diffraction patterns of as-deposited coatings of superconducting powders showed the presence of Y2O3 and other uncertain broad peaks, and there was a tendency that this broad peaks become dominant with increase of plasma power. When deposit was produced at 80volts (40KW), non of YBa2Cu3O7−x peak appeared, but the peak becomes dominant for 30volt (13.5KW) on the contrary. Those deposits less decomposed recover easily superconductivity by heat treatment for short time.

But in certain cases, no recovery was observed for those deposits. From these results, it was proved that plasma flame at high plasma power decomposed severely YBa2Cu3O7−x powders and where increase of arc voltage was more remarkable for the decomposition than that of arc current. This tendency was observed also in Tc measurement results of thermally treated coatings as shown in Fig. 2.

In the Fig. 3, similar results for the agglomerated powders were shown.

This figure shows that when the deposits were prepared by plasma spraying with the powders, the reaction among three kind of oxides became easier during plasma spraying with higher power than that with lower plasma power to give easily superconductive deposits by heat treatment in air. Results of EDX element analysis of as-deposited and thermally treated coatings are shown in Fig. 4.

From this figure, it can be seen that the uniform superconductive deposits were formed by heat treatment at 1223K for 2hr in air.
Table 1 Spray parameters and physical parameters of plasma jet.

<table>
<thead>
<tr>
<th>Torch</th>
<th>Arc current</th>
<th>Arc voltage</th>
<th>Plasma gas</th>
<th>Gas flows, S/D</th>
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<tr>
<td></td>
<td>9 MB</td>
<td>3 APG</td>
<td>9 MB</td>
<td>3 APG</td>
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<tr>
<td>Plasma gas</td>
<td>Ar</td>
<td>Ar/32 N2</td>
<td>Ar/32 N2</td>
<td>Ar/32 N2</td>
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<tr>
<td>Ato/Vol</td>
<td>450x20</td>
<td>500x20</td>
<td>500x20</td>
<td>450x20</td>
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<tr>
<td>Input power</td>
<td>12.9</td>
<td>18.3</td>
<td>25.6</td>
<td>27.7</td>
</tr>
<tr>
<td>Torque</td>
<td>7.9</td>
<td>5.6</td>
<td>5.6</td>
<td>3.0</td>
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<tr>
<td>Powder port</td>
<td>40-60</td>
<td>40-60</td>
<td>40-60</td>
<td>40-60</td>
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<tr>
<td>Orifice</td>
<td>38</td>
<td>38</td>
<td>38</td>
<td>38</td>
</tr>
<tr>
<td>Flow</td>
<td>4.0</td>
<td>5.0</td>
<td>3.0</td>
<td>5.0</td>
</tr>
<tr>
<td>velocity</td>
<td>12.9</td>
<td>16.2</td>
<td>16.2</td>
<td>16.2</td>
</tr>
</tbody>
</table>

Dc J/cc | 20.2(1) | 29.2(1) | 45.9 | 21.9 | 26.4 | 23.7(1) | 14.1(1) |

Roughly estimated parameters for comparing sprayed condition
Gas energy density: \( D_e = \frac{(Electricity - (Loss with cooling water))}{(Gas flow)} \)
Gas velocity: \( \nu = \frac{(Gas flow \times (cross-section of nozzle orifice))}{(electrically open circuit, appearance velocity)} \)

**Fig. 2** Effect of plasma power and thermal treatment on XRD patterns and Tc measurement results of coating (superconducting powders).

**Fig. 3** Effect of plasma power and thermal treatment on XRD patterns and Tc measurement results of coatings (agglomerated powders).
3.2 Effect of plasma gas on Tc of deposits

In case of superconducting powders, any deposits (as-deposited and thermally treated) did not exhibit \( \text{YBa}_2\text{Cu}_3\text{O}_{7-\delta} \) peak in X-ray diffraction patterns and superconductivity in Tc measurements, when deposits were produced by plasma spraying with both Ar/H\(_2\) and N\(_2\) plasma gas under plasma spraying conditions in Table 1.

From these results it can be considered that the heat supply to the powders during plasma spraying with Ar/H\(_2\) and N\(_2\) gas were more excessively decomposed the powders than that of Ar mono gas plasma spraying.

However, as shown in Fig. 5, for the agglomerated powders, Ar/H\(_2\) and N\(_2\) plasma gases facilitated the mutual reaction among oxides during plasma spraying to
give the deposits which showed the superconductivity by heat treatment for shorter time, as compared with Ar mono plasma gas as shown in Fig. 3. As making comparison between N₂ and Ar/H₂ gases, N₂ gas was more remarkable to react than Ar/H₂ gases because the deposit thermally treated for 0 min at 1223 K exhibited Tc = 83 K.

3.3 Effect of Gas flow rate and standoff distance on Tc of deposits

Above obtained results showed that it was necessary to control heat content of plasma jet in the case of the superconducting powders, in order to suppress decomposition of powders and keep the superconductivity of deposits.

The effect of standoff distance(S/D) on Tc of deposits was examined under plasma spray conditions, where heat content was controlled fully as shown in Table 1. The results were shown in Fig. 6. From this figure, it was seen that the decomposition of powders was suppressed more by plasma spraying at shorter distance of 90 mm. There was a presence of quite intensive YBa₂Cu₃O₇₋ₓ peaks in XRD patterns of both of as-deposited. Therefore, main peak of Y₂O₃ was greater than that of YBa₂Cu₃O₇₋ₓ for deposits at S/D = 115 mm. On the other hand, at S/D = 90 mm YBa₂Cu₃O₇₋ₓ peak remained greater than that of Y₂O₃. A reduction of resistance was observed from Tc measurement result of thermally treated deposit at 873 K for 6 hrs in the case of S/D = 90 mm and a resistance-temperature property like a sintered pellet was observed in the result of thermally treated deposit at 1223 K for 0 min. It may be considered that existence of YBa₂Cu₃O₇₋ₓ composition in deposits (preventing crystallization of Y₂O₃) through the deposition process facilitates to recover a superconductivity by simple thermal treatment. In other words, keeping particle in plasma jet for shorter time is an effective method of plasma spraying of superconducting powders (Fig. 5).

SEM photographs of surface and fracture surface of deposits obtained at S/D = 90 mm in Fig. 6 were shown in Fig. 7. These show that as-deposited is not seen as a partially layered structure constituted with flattened particles, slight melting of deposit was observed by heat treatment at 873 K, and crystallized grains fully were grew up by heat treatment at 1223 K.

Plasma spraying parameters in this examination are shown in Table 1. For comparing sprayed conditions, roughly estimated parameters of gas energy density (DE) and gas velocity (v) are introduced. In the case of superconducting powders, high Tc after thermal treatment was obtained under the condition of 24 J/cc (DE). However at 500 A × 40 Volts, Ar/H₂ and N₂, high Tc was not obtained. It may be considered that high gas velocity is effective for obtaining high Tc deposits in insufficient melting of particles and prevent decomposition of those. On the other hand, high heat content results in a excessively decomposition of particles and no recovery of superconductivity by thermal treatment. Because high Tc by thermal treatment of a very short period was obtained when deposits were produced by plasma spraying under the conditions of high velocity, low DE and short standoff distance where the decomposition of particles was controlled. In the case of agglomerated powders, high DE is not almost necessary because best result is obtained with N₂ plasma gas when DE is not so great, and all thermally treated deposits for several hours showed reliably a good superconducting property, so that this processing is useful if deposits are thermally treated at high temperature. In other word, as a result of this examination, it was proved that N₂ gas is more suitable for in-flight composition than Ar or Ar/H₂ gas for air plasma spraying.

4. Conclusion

The effect of plasma spray parameters such as plasma power, plasma gas, gas flow rate and standoff distance on
property and structure of YBa$_2$Cu$_3$O$_{7-x}$ coatings was examined by using superconducting powders and agglomerated powders of Y$_2$O$_3$, 2BaCO$_3$ and 3CuO.

The results obtained were summarized as follows.

(1) In order to obtain superconducting coatings uniformly and easily by post heat treatment, it was important to suppress the decomposition of superconducting powders of YBa$_2$Cu$_3$O$_{7-x}$ during plasma spraying. Therefore it was necessary to select plasma spray parameters of lower heat content and adopt lower plasma power, higher gas flow rate and etc.

(2) For the agglomerated powders, it was important to facilitate the mutual reaction among three kinds of oxides of Y$_2$O$_3$, Ba$_2$CO$_3$ and CuO during plasma spraying. So it was necessary to use N$_2$ plasma gas in order to increase heat content.

References


